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FINAL SUMMARY REPORT PROGRAM FOR THE DEVELOPMENT OF HIGH TEMPERATURE ELECTRICAL MATERIALS AND COMPONENTS

by

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16. Abstract

This final report summarizes data and technical analyses presented in three reports on electrical materials intended for advanced space electric power systems. Three major subjects were investigated under NAS3-10941 as follows:

1. Evaluation of high temperature, space-vacuum performance of selected electrical materials and components.

2. High temperature capacitor development.

3. Evaluation, construction, and endurance testing of compression sealed pyrolytic boron nitride slot insulation.

The first subject above covered the aging and evaluation of electrical devices constructed from selected electrical materials under a previous program (NAS3-6465). Individual materials performances were also evaluated and reported. The second subject included study of methods of improving electrical performance of pyrolytic boron nitride capacitors. The third portion was conducted to evaluate the thermal and electrical performance of pyrolytic boron nitride as stator slot liner material under varied temperature and compressive loading. Conclusions and recommendations from this program are presented.

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FOREWORD

The work reported herein was sponsored by the NASA Lewis Research Center. Mr. R. A. Lindberg of NASA was the Project Manager for this program. Mr. T. A. Moss formerly of NASA is also acknowledged for his interest and support of this work.

The Westinghouse Electric Corporation, Aerospace Electrical Division (WAED) was responsible for the overall technical direction of the program. Westinghouse Research and Development Center was a subcontractor in the program. Other subcontractors were Westinghouse Advanced Reactors Division, Westinghouse Lamp Division, and the EIMAC Division of Varian Associates. The work was performed in execution of this contract during the time period from April 1968 to November 1969.

Detailed reporting of the work accomplished on this program is given in three Topical Reports entitled "Evaluation of High Temperature Electrical Materials and Components" (NASA CR-72850, WAED 70.08E), "High Temperature Capacitor Development" (NASA CR-1799, WAED 69.29E), and "Evaluation, Construction and Endurance Testing of Compression Sealed Pyrolytic Boron Nitride Slot Insulation" (NASA CR-72849, WAED 69.26E).

In a project of this magnitude, many skilled engineers and scientists are consulted. No attempt will be made to single out a person's specific contribution, since in many cases, it was in several areas. Those who contributed throughout all or much of the extensive program are recognized below:

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SUMMARY

This is the Final Report on NAS3-10941 which summarizes over 309 pages of data and technical analyses presented in three reports on electrical materials for advanced space electric power systems.

The areas investigated included three major subjects as follows:

1. Evaluation of high-temperature, space-vacuum performance of selected electrical materials and components.

2. High temperature capacitor development.

3. Evaluation, construction, and endurance testing of compression sealed pyrolytic boron nitride slot insulation.

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SECTION I

INTRODUCTION

This is the final report on NASA Contract NAS3-10941 summarizing the program results and technical developments yielded from high-temperature, space-vacuum exposure tests and post-exposure evaluations of both electrical devices and their component materials, high temperature capacitor development, and compression-sealed, pyrolytic boron nitride slot insulation development. The contract consisted of three programs as follows:

Program I - Evaluation of High Temperature Electrical Materials and Components.

Program II - High Temperature Capacitor Development.

Program III - Evaluation, Construction and Endurance Testing of Compression Sealed Pyrolytic Boron Nitride Slot Insulation.

Program I completed the aging and evaluation of electrical devices constructed from selected electrical materials under a previous program (ref. 1). The performances of a generator-type stator containing a potassium-loaded ceramic bore seal, a transformer, and two solenoids were reported and evaluated. The materials used in the above devices were also evaluated both for individual performances and as parts of the materials system.

Program II continued the development work begun in an earlier program (ref. 2) on high-temperature pyrolytic boron nitride capacitors. Methods of improving electrical performance were studied, resulting in greater capacitance stability and lower electrical losses.

Program III was conducted to evaluate the performance of pyrolytic boron nitride as a stator slot liner material. Clearances and compressive loadings were varied with temperature and slot liner heat conductance was measured from the electrical conductor to the enclosing statorette under the varied conditions.

Detailed reporting of the work accomplished on these programs is given in three reports entitled "Evaluation of High Temperature Electrical Materials and Components" (NASA CR-72850, WAED 70.08E)(ref. 3), "High Temperature Capacitor Development" (NASA CR-1799, WAED 69.29E)(ref. 4), and "Evaluation, Construction and Endurance Testing of Compression Sealed Pyrolytic Boron Nitride Slot Insulation" (NASA CR-72849, WAED 69.26E)(ref. 5). Four Quarterly Reports (refs. 6, 7, 8 and 9) were also written to report progress as the programs were carried out.

Sections II, III, and IV summarize the technical results on evaluation of electrical materials and components (Program I), capacitor development (Program II), and compression sealed pyrolytic boron nitride slot insulation (Program III). Each section contains a technical discussion and conclusions and recommendations for that program.

SECTION II

PROGRAM I - EVALUATION OF HIGH TEMPERATURE ELECTRICAL MATERIALS AND COMPONENTS

SUMMARY OF TECHNICAL RESULTS

The objectives of this program were to complete additional thermal-vacuum endurance testing of the ac stator with bore seal and, after completion of endurance testing of the components, to evaluate component performances and individual and interacting performances of the materials used in the ac stator and bore seal, transformer, and two solenoids.

The program was divided into four main areas of concentration as follows:

1. Completion of 10,000 hours endurance testing of the ac stator and bore seal at 1300° F in a 10^{-9} torr range environment.
2. Evaluation of component performance for the thermal-vacuum endurance-tested ac stator, bore seal, transformer, and two solenoids.
3. Evaluation of materials performances, both individually and as members of a materials system.
4. Evaluation of performance of thermal-vacuum test chambers.

Five thousand hours endurance testing of the ac stator and bore seal at 1300° F and 10^{-9} torr range was completed. This exposure of these units was in addition to a previous 5000 hours endurance testing at the same conditions (ref. 1). Heat to maintain the stator hot-spot temperature of 1300° F was obtained from two sources. During the endurance test, the stator was supplied with three-phase, 400-Hz power with a potential of approximately 485 volts between phases. The current in each phase was maintained at 50 amperes to induce Joule (I^2R) heating in the conductors. The remainder of the heat required was supplied by a heating element which was an integral part of the test chamber. Electrical data were taken each week during the endurance test to monitor conductor and insulation system performance. Figure 1 is a plot of dc insulation resistance values, phase-to-phase and phase-to-ground, as a function of time for the 10,000 hour test period. A gradual improvement in insulation performance is evident as indicated by the estimated trend lines. The ac stator improved in all electrical aspects during the exposure.

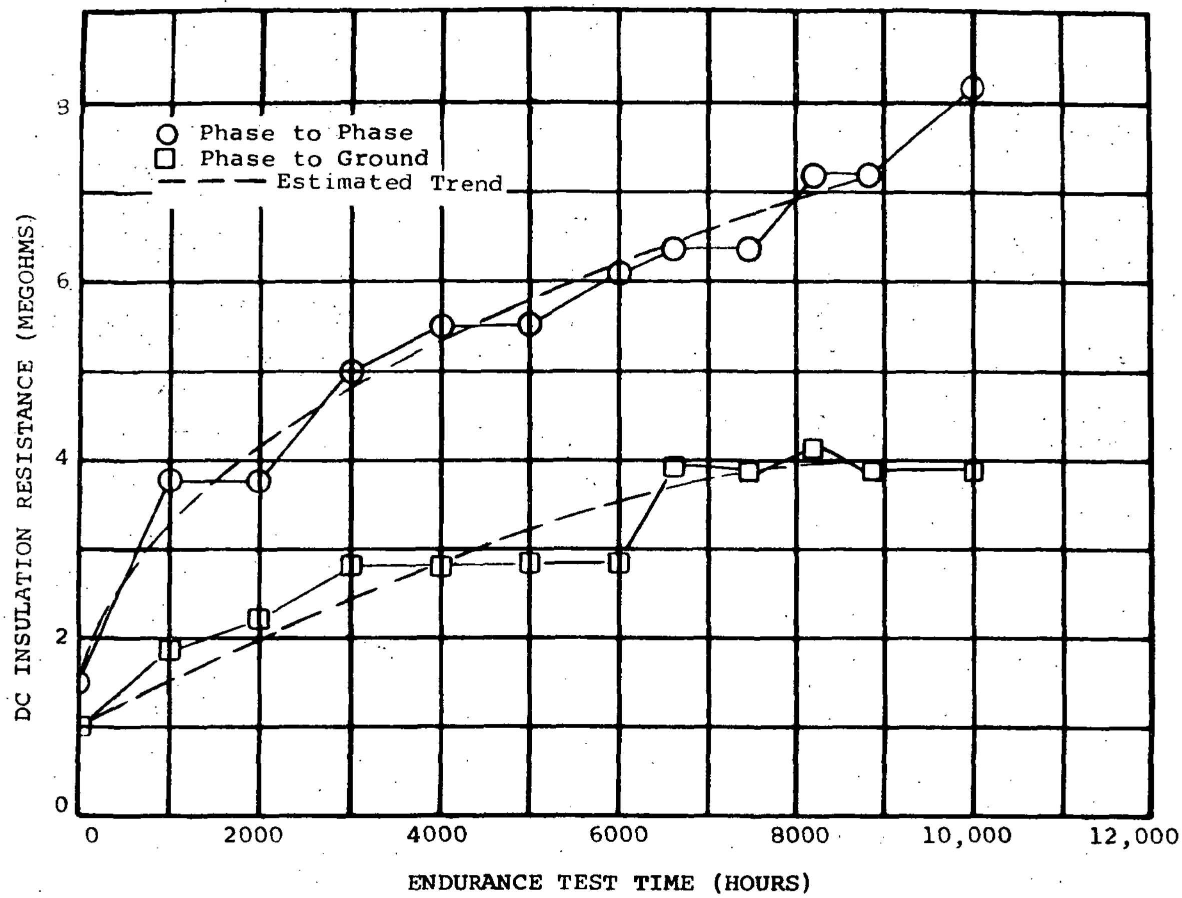

Figure 1. - Stator DC Insulation Performance as a Function of Endurance Test Time in Ultrahigh Vacuum with 1300° F Hot-Spot (Slot) Temperature. Five Hundred Volts DC was Applied Between Phases and Between Each Phase to Ground.

After the exposure period was concluded and final electrical tests at temperature were completed, the thermal-vacuum chambers were cooled and opened. Figure 2 is a view of the chamber interior with top heat shields removed showing the stator and the bore seal capsule. No evidence of potassium leakage was noted as the stator and bore seal capsule were removed from the test chamber. Figure 3 is a photograph of the bore seal capsule and support pedestal after removal from the test chamber. The capsule outer diameter displayed stain patterns which replicated appearances of the stator laminations, slot wedges, and winding end turns. The source of these stains was determined as part of the post-test evaluation. Table 1 shows the analytical data

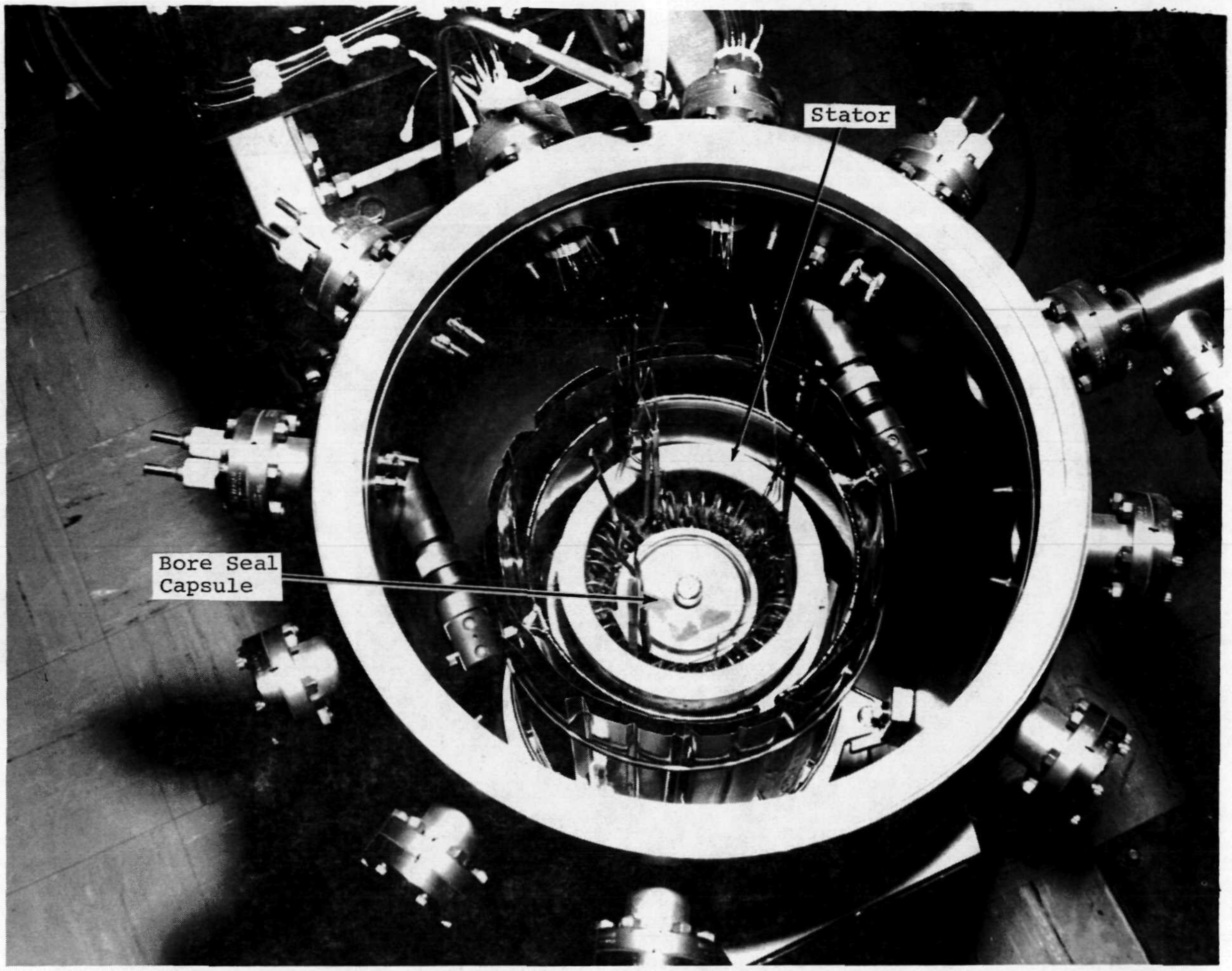

Figure 2. - Photograph of the Stator and Bore Seal Capsule in the Test Chamber After Removal of the Top Heat Shield and After the 10,000-Hour, 1300° F Ultrahigh Vacuum Endurance Test.

Figure 3. - Photograph of the Bore Seal Capsule and Support Pedestal After the 10,000-Hour Potassium Exposure Test at 1300° F in Vacuum.

Table 1. - Emission Spectrochemical Analysis of Deposits From the Bore Seal Capsule Exterior After 10,000 Hours Exposure in the Stator at 1300° F at a Pressure in the 10^{-9} Torr Range.

Sample	Major Constituents (>10 percent of deposit)	Minor Constituents (<10 percent of deposit)
Black Deposit - Center Periphery	Fe, Mn	Ti, B, Co, Ni, Zr, V, Cu
Light Gray Deposit - Bottom Periphery	Fe, Ni	Zr, Mo
Dark Gray Deposit - Bottom Periphery	Fe, Ni	Zr, Mn, V

identifying the stains. The bore seal capsule was radiographed to determine location of the potassium and the location of possible internal flaws. No flaws were detected in any component of the bore seal capsule. The potassium was removed with due care exerted to prevent contamination and analyzed as reported in table 2. These data indicate good stability of the components of the capsule system. The capsule was subsequently cleaned by thermal-vacuum baking at 1832° F and 2×10^{-7} torr for twenty minutes. The beryllia ceramic capsule walls were cut into pieces which were prepared as modulus-of-rupture bar specimens. Comparison of unaged and aged flexural strength values (ref. 1) demonstrated no penetrating effects due either to vacuum or potassium exposure. The bore seal capsule brazed joint specimens were sectioned and analyzed by electron microprobe, visual microscopy, and microhardness survey. The specimens were examined for evidence of potassium penetration and evidence of material degradation or incompatibility. The condition of the capsule including the brazed joints was good, indicating material stability.

The ac stator was partially disassembled for examination of constituent parts. The stator magnetic laminations were

Table 2. - Metallic Impurities, By Spectrographic Analysis, of 10,000-Hour, 1300° F Exposed Bore Seal Capsule Potassium and Process Control Potassium Chlorides.

Impurity (parts per million by weight = micrograms of element per gram of potassium ± 30 percent relative based on potassium)

Element	Potassium as Loaded in Bore Seal(a)	Base Potassium Chloride(b)	Processed Potassium Chloride(b)	Bore Seal Potassium After Exposure and Processing(b)	Calculated Bore Seal Potassium Impurity Increase After 10,000-Hour Exposure
Ag	1	<0.5(e)	<0.5(e)	<0.5(e)	<0.5(e)
Al	3	9	30	150	117
B	<5	<0.5(e)	0.5	2.6	2.1
Ba	<3	(f)	(f)	(f)	(f)
Be(c)	<1	<0.5(e)	<0.5(e)	22	22
Bi	(f)	<5(e)	<5(e)	<5(e)	<5(e)
Ca	4	10	125	190	61(d)
Cb	(f)	<10(e)	<10(e)	21	21
Cd	(f)	<50(e)	<50(e)	<50(e)	<50(e)
Co	<5	<10(e)	<10(e)	<10(e)	<10(e)
Cr	<1	<20	<20	<20	<20
Cu	<1	6	21	58	37
Fe	5	4	81	92	6
Ge	(f)	<20(e)	<20(e)	<20(e)	<20(e)
In	(f)	<20(e)	<20(e)	<20(e)	<20(e)
Mg(d)	<1	<2(e)	20	27	7
Mn	<1	<0.5(e)	3.8	7.2	3.4
Mo	<5	<2(e)	<2(e)	<2(e)	<2(e)
Na	3	(f)	(f)	(f)	(f)
Ni	<1	<5(e)	6	19	13
Pb	<1	<10(e)	17	16	<10
Pt	(f)	<20(e)	<20(e)	440	440
Rh	(f)	<5(e)	<5(e)	950	950
Si	10	<6	19	41	12
Sn	<1	<6(e)	<6	7	7
Sr	<1	<0.5(e)	<0.5(e)	<0.5(e)	<0.5(e)
Ti	<5	<3(e)	4	3	<3
V(c)	<1	<2(e)	<2(e)	<2(e)	<2(e)
Zr(c)	<10	<5(e)	<5(e)	<5(e)	<5(e)

(a) Batch analyses supplied by Mine Safety Applied Research, Evans City, Pa.
(b) Spectrographic analyses by Westinghouse Power Systems, Waltz Mill Site, Madison, Pa.
(c) These are the major elements specified for analysis.
(d) The indicated quantities of these elements are not considered to be significant.
(e) < means less than, and indicates the limit of detection for the element, or a limit due to contamination.
(f) Not determined.

examined for coercive force, microhardness, brittleness, metallurgical structure, and interstitial analysis. Substantial grain growth and hardness increase were noted. The magnetic quality of the iron-27% cobalt alloy improved slightly after the thermal-vacuum endurance testing. This improvement was attributed to the magnetic annealing effect that was created by the ac magnetic field induced during the energized exposed at elevated temperature.

The magnetic laminations were embrittled by oxide diffusion along the grain boundaries. The source of the second-phase oxygen was believed to be adherent surface oxides present after lamination and stator processing.

The Inconel-clad silver conductors were examined visually, electrically and by electron microprobe. All results indicated good stability. Figure 4 shows electron microprobe scans on cross-section specimens of the unaged and aged conductor in which the lack of diffusion is notable. The distances of nine and eleven microns noted in the chromium scans are within the limits of experimental error for this analytical method.

The stator conductor electrical insulation was examined by visual microscopy, spectrographic analyses of unaged and aged specimens, and by electrical tests. Some minor chemical changes were reported. Electrical data demonstrated gradual improvement throughout the test exposure as shown in part by figure 1.

The rigid electrical insulation components were electrically and mechanically stable. Some small localized chemical changes were evident as variously colored spots which apparently had no effect upon operating properties.

A transformer and two solenoids were endurance-tested for 5000 hours at 1300° F in high vacuum on a previous program (ref. 10). Reference 10 contains the design, test details and electrical test results. During the 5000-hour endurance test, the transformer primary (high voltage) winding experienced a failure after 244 hours at 1300° F. After failure, the test was continued with 600 volts ac applied across each winding to ground, to investigate the effects of long term voltage stress on insulation performance. Table 3 gives the transformer endurance test and post-endurance test electrical performance data at room temperature and ambient pressure. The extremely high post-endurance test conductor resistance was caused by a separation within the winding associated with the primary winding failure. DC insulation resistance displayed a tendency toward improvement with aging while ac leakage increased a small amount. Aluminum oxide rigid electrical insulation components and boron nitride flexible sheet insulation performed well, both individually and in combination. The electrical conductors were stable

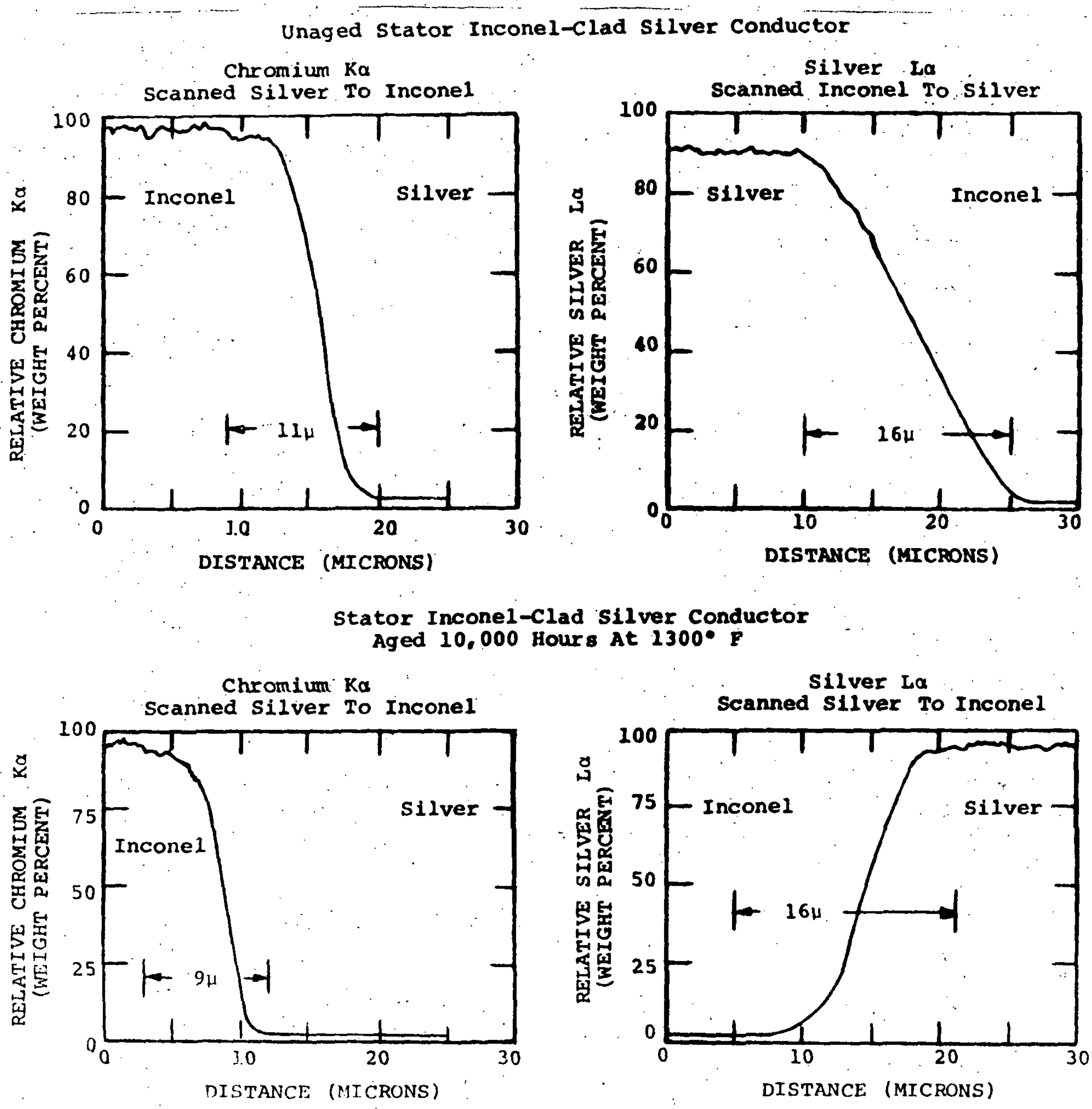

Figure 4. - Electron Microprobe Scans For Silver and Chromium in Aged (10,000 Hours at 1300° F Ultrahigh Vacuum) and Unaged Stator Inconel-Clad Silver Conductors.

Table 3. - Comparison of Transformer Room Temperature Bench Test Electrical Data Taken Before and After a 5000-Hour Thermal Vacuum Exposure.

MEASUREMENT	TRANSFORMER WINDING (a)	BEFORE ENDURANCE TEST		AFTER ENDURANCE TEST	
		OHMS	TEMPERATURE (°F)	OHMS	TEMPERATURE (°F)
Conductor Resistance	Primary[b]	1.7640	72	3×10^9	78
	Secondary	0.0080	72	0.00805	78
		MEGOHMS @ 500 Vdc	TEMPERATURE (°F)	MEGOHMS @ 500 Vdc	TEMPERATURE (°F)
DC Insulation Resistance	Primary to Secondary[b]	7×10^4	72	3.5×10^4	78
	Primary to Ground	1.45×10^4	72	4.3×10^4	78
	Secondary to Ground	5.5×10^4	72	8.0×10^4	78
		MICROAMPS @ 500 Vac	TEMPERATURE (°F)	MICROAMPS @ 500 Vac	TEMPERATURE (°F)
AC Leakage Current	Primary to Secondary[b]	<1	72	14	78
	Primary to Ground	<1	72	9	78
	Secondary to Ground	<1	72	9	78

(a) Transformer windings are designated as primary (high voltage) and secondary (low voltage) for winding conductor resistance measurements.

(b) Primary (high voltage) winding failed after 244 hours at test temperature (1300°F hot spot).

except in the primary winding at the malfunction site. At that location, the silver had melted and penetrated out through the Inconel cladding, producing the aforementioned high resistance. The magnetic laminations were satisfactory electrically and magnetically but were embrittled as in the ac stator.

Two solenoids were endurance-tested in the same chamber with the transformer. One solenoid was continuously energized during the test except as required for taking weekly electrical performance readings. The second solenoid was intermittently energized during the test period for weekly electrical performance readings. The energized solenoids supported a 3.0-pound weight during the test exposure period. Table 4 compares pretest and post-endurance test room ambient environment electrical performance data for the two solenoids. These data show good endurance for the materials and the devices. The continuously energized solenoid operated without degradation through 4899 hours of the 5000-hour thermal endurance test. The interruption in operation was caused by a short circuit between the external leads and was not related to the device. The intermittently energized solenoid functioned throughout the 5000-hour thermal endurance test in a 10^{-9} torr range environment.

The two thermal-vacuum test chambers used on this and the previous program (NASA Contract NAS3-6465) provided satisfactory service without maintenance for nearly 20,000 hours accumulated vacuum operating time. Each chamber was of the "cold wall" construction with water cooling and included tantalum resistive heating elements and heat shields, a 500-liter-per-second sputter ion pump, a tungsten-filament-heated titanium sublimation pump, and associated power supplies, controls, and instrumentation. Reference 3 contains detailed discussion of operating history and post-endurance test conditions plus residual gas analysis data.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1. A high-temperature stator and bore seal containing potassium exhibited satisfactory functional performance for 10,000 hours at 1300° F in ultrahigh vacuum.

2. Stator materials of construction, including iron-27% cobalt magnetic laminations and solid forgings, Inconel-clad silver conductor; refractory filled glass-bonded "S" glass conductor insulation and high purity alumina ($>99\%Al_2O_3$) rigid insulation, all performed without functional degradation. Improvement with aging was found in magnetic materials (lowered

Table 4. - Comparison of Solenoid Room Temperature Bench Test Electrical Data Taken Before and After a 5000-Hour Endurance Test in Ultrahigh Vacuum with a 1300° F Hot-Spot Temperature.

MEASUREMENT[a]	SOLENOID ENERGIZED	BEFORE ENDURANCE TEST			AFTER ENDURANCE TEST		
		OHMS		TEMPERATURE (°F)	OHMS		TEMPERATURE (°F)
Conductor Resistance	Continuously	12.05		72	12.53		78
	Intermittently	11.65		72	12.05		78
		MEGOHMS @ 500 Vdc		TEMPERATURE (°F)	MEGOHMS @ 500 Vdc		TEMPERATURE (°F)
DC Insulation Resistance	Continuously	4×10^4		72	8.0×10^4		78
	Intermittently	1.05×10^4		72	7.0×10^4		78
		MICROAMPS @ 500 Vac		TEMPERATURE (°F)	MICROAMPS @ 500 Vac		TEMPERATURE (°F)
AC Leakage Current	Continuously	1		72	2.0		78
	Intermittently	1		72	2.0		78
		VOLTS DC	AMPS DC	TEMPERATURE (°F)	VOLTS DC	AMPS DC	TEMPERATURE (°F)
Load Performance							
Minimum Pickup	Continuously	5.0	0.4	72	4.9	0.38	78
Minimum Pickup	Intermittently	4.8	0.395	72	6.0	0.46	78
Minimum Hold	Continuously	0.6	0.05	72	0.6	0.05	78
Minimum Hold	Intermittently	0.8	0.07	72	0.7	0.06	78

(a) In a single-winding component, measurements can only be made within the winding (conductor resistance, volts, amps) or from winding to ground.

coercive force due to magnetic annealing) and the insulation system improved (increased dc resistance and decreased ac leakage current) with aging due to outgassing.

3. The bore seal, consisting of 99.8% beryllia ceramic brazed to columbium-1% zirconium end pieces with a 60Zr-25V-15Cb alloy, remained potassium leak tight after 10,000 hours testing at 1300° F.

4. The flexural strength of 99.8% beryllia ceramic remained unchanged after potassium exposure at 1300° F for 10,000 hours.

5. Two high-temperature solenoids performed satisfactorily after 5000 hours at 1300° F in ultrahigh vacuum.

6. Solenoid materials of construction, including iron-27% cobalt forgings, high purity ($>99\% Al_2O_3$) alumina components, Inconel-clad silver conductor, refractory filled glass-bonded "S" glass conductor insulation, and boron nitride fiber cement, functioned satisfactorily.

7. A high-temperature transformer performed satisfactorily for 244 hours prior to a failure in the primary winding. Failure was attributed to a turn-to-turn short circuit. Possible causes for lack of electrical insulation at the failure are: (1) abrasion of insulation due to relative motion caused by opposing magnetic forces, and (2) a pin-hole in the Inconel cladding that allowed migration of silver into the insulation.

8. Iron-27% cobalt laminations, high purity ($>99\% Al_2O_3$) alumina insulation, boron nitride fiber cement, and boron nitride fiber paper performed satisfactorily in the transformer.

9. Boron nitride fiber paper, used as insulation between winding layers, performed satisfactorily. Post aging evaluation revealed satisfactory compatibility with the fibrous conductor insulation.

10. Since the transformer experienced a fault, the long-term performance of a transformer with the designated materials system could not be assessed.

11. Two ion-pumped thermal ultrahigh vacuum chambers performed flawlessly for 19,700 hours at a pressure in the 10^{-9} torr range or lower.

12. Although a light deposit of transported material was visible on the external surfaces of all components after completion of testing in vacuum for periods as long as 10,000 hours, the performance of components was not degraded.

13. Iron-27% cobalt alloy, especially in the thin lamination form was found to be brittle after aging for 5000 or 10,000 hours at 1300° F. This brittleness is not a problem in the static applications but dynamic or highly stressed applications should be carefully considered until the mechanism of embrittlement is understood and mitigated.

14. The materials used in the high-temperature stator, solenoids and transformer exhibited low outgassing levels at 1300° F at pressures in the 10^{-9} torr range. Outgassing of the insulation system during the initial test period improved insulation performance.

Recommendations

1. A method of non-destructive testing of clad silver conductors should be developed and applied to reliable high temperature space electric power systems. The test method should be capable of detecting flaws including pinholes in the cladding. Detection and elimination of such flaws by 100 percent inspection of the conductor would prevent silver migration at elevated temperatures.

2. The glass-bonded fiber glass conductor insulation should be improved to enhance mechanical strength and handling characteristics and improve adherence in the cured condition.

3. An improved, high-strength potting compound is required for electrical apparatus for applications above 1000° F.

4. Thorough mechanical testing of electrical materials should be performed after long-term thermal vacuum exposure to define material limitations.

5. It is recommended that dynamic tests of a high-temperature alternator be started to determine mechanical and power characteristics under load.

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SECTION III

PROGRAM II - HIGH TEMPERATURE CAPACITOR DEVELOPMENT

SUMMARY OF TECHNICAL RESULTS

The purpose of Program II was to improve the high temperature stability of pyrolytic boron nitride capacitors which were developed under NASA contract NAS3-6465 (ref. 2). Pyrolytic boron nitride capacitors were made by sputtering thin film electrodes on the wafer surfaces. Vacuum tests showed that these capacitors had a voltage breakdown of 7000 volts per mil at 1100° F, a capacitance change from room temperature to 1100° F of about 1.7 percent and a dissipation factor (tan δ) of less than 0.001 at 1100° F. A five-wafer stack of these capacitors which was continuously energized at voltages up to 1000 volts dc per mil for over 1000 hours at 1100° F showed a capacitance change of less than three percent. This change in capacitance was attributed to a slight separation of electrodes from the pyrolytic boron nitride surfaces.

Program II of this contract is reported in reference 4. That report presents the results of two processing methods that substantially improved capacitor stability and overall electrical properties underaging and thermal cycling conditions. As preliminary effort to the two approaches, electrical properties were determined for pyrolytic boron nitride in several surface conditions. Data representative of this step is presented in table 5.

One method investigated was surface texturizing of pyrolytic boron nitride wafer surfaces by radio frequency (rf) off-sputtering. Removal of 3000 angstroms of surface material before application of electrodes resulted in a two- to three-fold reduction in electrical losses. Surface texture (ratio of true to apparent or geometric area) was measured by a "double layer capacitance technique". These data were correlated with electrode adherence and capacitor losses and stability under thermal cycling and aging conditions at 1300° F and 1100° F in vacuum ($< 1 \times 10^{-6}$ torr).

The second method that was studied was the deposition of a sputtered boron nitride barrier layer to a portion of the outer surfaces of the electrodes. Figure 5 shows a schematic illustration of an application of the above technique. Table 6 presents aging test data for capacitors with and without sputtered

Table 5. - Data Comparing Properties of Single-Wafer Capacitors Made with New and Old Lots of Pyrolytic Boron Nitride

Group	Pyrolytic Boron Nitride Lot[a]	Wafer Surface Finish	Platinum Electrode Area (in^2)	Calculated Wafer Thickness (inches)	Capacitance (pF)[b] and Dissipation Factor (tan δ) at Room Temperature, 1 kHz, C = pF	1 kHz, tan δ	10 kHz, C = pF	10 kHz, tan δ	Comments
A	1	Matte	0.379	0.0008	353.91	0.00079	353.53	0.00060	Made on NAS3-10941
	1		0.379	0.0008	360.53	0.00102	360.02	0.00091	
	1		0.379	0.0007	395.88	0.00098	395.26	0.00065	
	1		0.379	0.0012	246.71	0.00119	246.33	0.00096	
	2		0.379	0.0010	287.61	0.00116	287.21	0.00097	Made on NAS3-10941
	2		0.364	0.0009	326.17	0.00094	325.75	0.00085	Made on NAS3-6465
	2		0.364	0.0010	277.86	0.00127	277.39	0.00108	
	2	Matte	0.364	0.0010	269.19	0.00071	268.93	0.00071	
B	1	Polished	0.217	0.0012	230.48	0.00075	230.25	0.00060	Made on NAS3-10941
	1		0.217	0.0009	182.12	0.00076	181.96	0.00058	
	2		0.364	0.0006	454.58	0.00061	454.23	0.00054	Made on Nas3-6465
	2		0.364	0.0004	657.28	0.00079	656.63	0.00072	
	2	Polished	0.364	0.0005	566.69	0.00108	565.90	0.00105	

(a) Lot identification refers to Boralloy, pyrolytic boron nitride plate material (Carbon Products Division, Union Carbide Corporation, 270 Park Ave., N.Y.C., N.Y.). Lot 1 was purchased on order 39-J-387991-CC and received August 24, 1965. Lot 2 was purchased on order 39-401673 and received January 11, 1967. Material specifications are in accordance with the manufacturer's quality control standards for Boralloy.

(b) Units of capacitance are in picofarads.

diffusion layers. The effects of compression on electrical properties of pyrolytic boron nitride capacitors were also studied. Compressive force effects on dissipation factor at various temperatures are shown in figure 6.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1. The effectiveness of a radio frequency-sputtered boron nitride barrier layer on the platinum electrodes of a pyrolytic boron nitride capacitor was demonstrated. Capacitors subjected to these tests showed that the barrier layers prevented interelectrode diffusion bonding between electrodes on adjacent wafers.

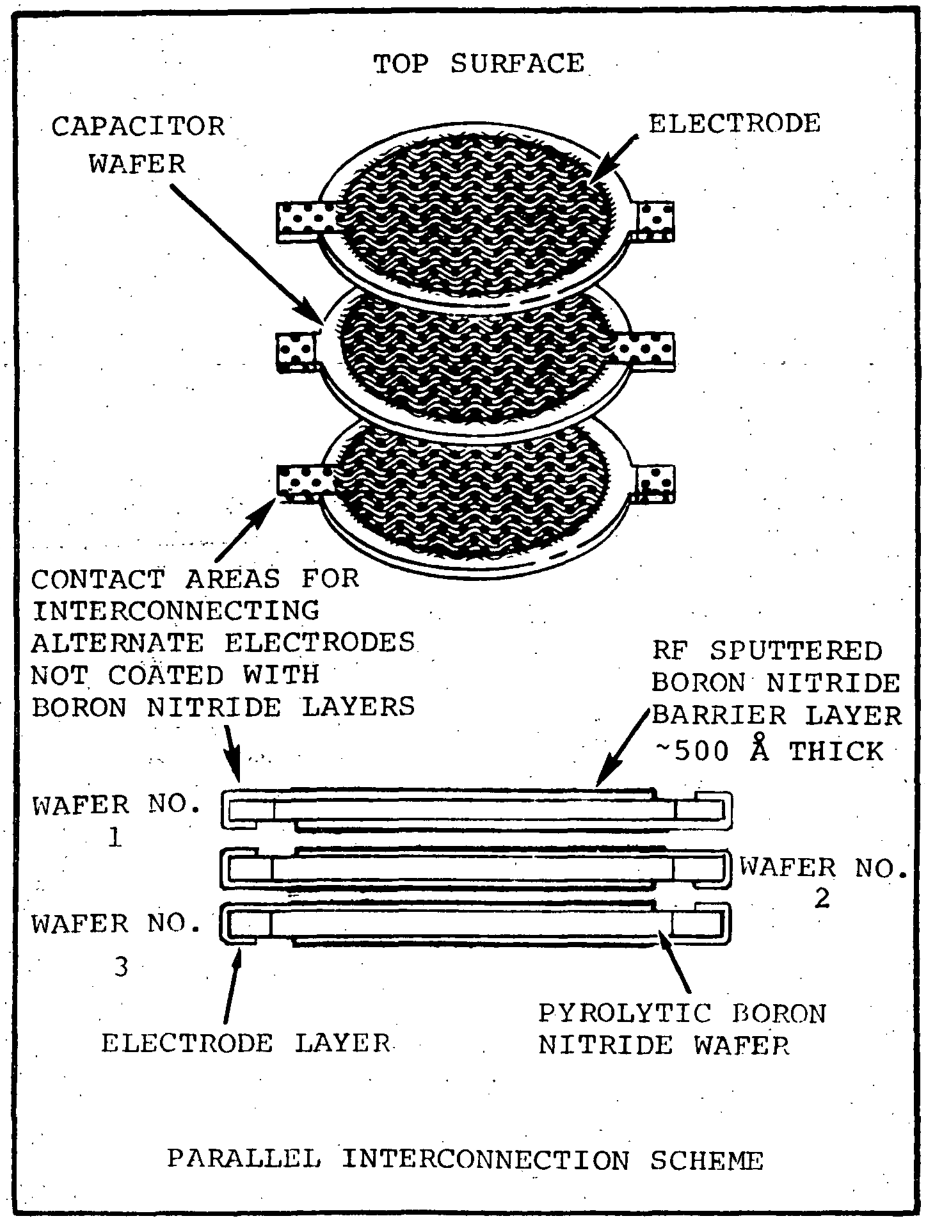

Figure 5. - A Representation of the Three-Wafer Tabbed Capacitor Showing Location of Radio Frequency Sputtered Boron Nitride Barrier Layers on Top and Bottom Electrode Surfaces of Each Capacitor Wafer

Table 6. - Aging Test Data for Pyrolytic Boron Nitride Capacitors With or Without Sputtered Diffusion Barrier Layers

Elapsed Time With 500 VDC Applied	Furnace Temperature (°F)	Pressure (torr)	Capacitance (pF)(c) and Dissipation Factor (tan δ)				ΔC/C$_o$ x 100 at 1 kHz	RC Product DC Resistance (MΩ) x Capacitance (μF)
			1 kHz		10 kHz			
			C = pF	tan δ	C = pF	tan δ		
DATA FOR THREE WAFER CAPACITOR WITH DIFFUSION BARRIER LAYERS								
Room Temp.	~80	1×10^{-6}	883.27	0.000192	883.08	0.00025	--	8.8×10^{3}
Start	1100	9×10^{-8}	861.73	0.0024	858.78	0.0018	--	--
21 hrs.	1090	9×10^{-8}	861.56	0.0037	858.02	0.0021	--	11.0
50 hrs.	1100	8×10^{-8}	861.46	0.0044	857.38	0.0024	-0.031%	9.0
--	~100	7.9×10^{-8}	875.01	0.000087	874.95	0.00015	--	4.9×10^{4}
DATA FOR FIVE WAFER CAPACITOR - NO DIFFUSION BARRIER LAYERS (NAS3-6465)								
Room Temp.	~80	2.6×10^{-7}	1422.85	0.00058	1421.75	0.00064	--	2.4×10^{5}
Start	1111	4×10^{-7}	1381.39	0.00231	1378.77	0.00119	--	14.7
65 hrs.	1113	2×10^{-8}	1366.84	0.00267	1363.30	0.00145	-1.05%	17.1
--	~80	5.3×10^{-9}	1363.69	0.00117	1361.67	0.00131	--	1.06×10^{3}

(a) Chromel/Alumel thermocouple located approximately 2 inches from test specimen.

(b) C_o = capacitance value at 1 kHz when 500 VDC is initially applied.
$\Delta C = C_o = C_T$; where C_T = capacitance value at 1 kHz after specified elapsed time with 500VDC applied continuously.

(c) Units of capacitance are in picofarads.

2. Pyrolytic boron nitride capacitor losses (dissipation factor) at 1100° F can be reduced three-fold by radio frequency off-sputtering or surface texturizing pyrolytic boron nitride wafer surfaces prior to the deposition of sputtered platinum electrodes. This effect is attributed to the removal of the mechanically disturbed surface layers produced during final lapping and subsequently removed during off-sputtering.

3. Surface texturizing of pyrolytic boron nitride capacitor wafers by mechanical and off-sputtering methods have shown a surface area ratio of 6 to 1 (actual to apparent). It appears that the surface area of off-sputtered pyrolytic boron nitride wafers reach a constant value with off-sputtering time, irrespective of the initial surface texture (smooth or mechanically roughened) of the wafer.

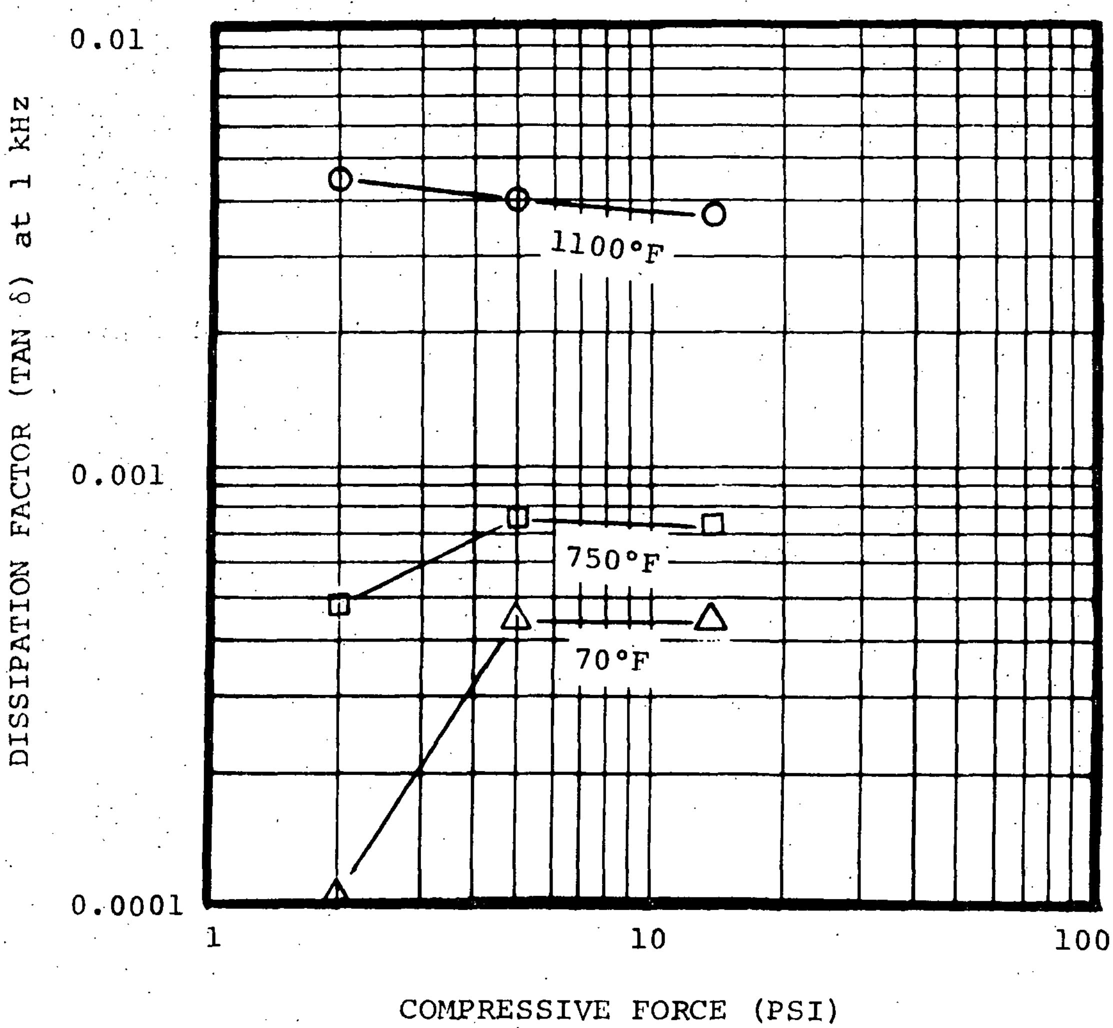

Figure 6. - Effect of Compression on Dissipation Factor at Room Temperature, 750°, and 1100° F in Vacuum (1 x 10^{-6} torr range).

4. Adherence tests conducted on platinum electrodes which had been sputtered onto texturized pyrolytic boron nitride capacitor wafers showed bond strengths to 1000 psi. In contrast, the bond strength of platinum to polished pyrolytic boron nitride wafers was too low to determine.

5. Electrical characterization tests were performed on several single wafer capacitors fabricated from a new lot of pyrolytic boron nitride. The overall results indicate there are no intrinsic differences in the electrical properties of the new lot of material when compared to the material used on an earlier program (NAS3-6465).

6. Pyrolytic boron nitride capacitors with platinum electrodes subjected to fifty thermal cycles between 300° and 1300° F and then aged for fifty hours at 1100° F in vacuum showed no significant changes in capacitance and dissipation factor.

7. Three pyrolytic boron nitride capacitors with sputtered gold electrodes were subjected to 64 thermal cycles between 300° and 1100° F. Substantial increases in dissipation factor in two out of three of these devices indicate that gold electrodes on pyrolytic boron nitride capacitors are less stable than platinum electrodes.

Recommendations

1. Pyrolytic boron nitride capacitor wafers that are mechanically lapped to final thickness should be pre-conditioned by radio frequency off-sputtering wafer surfaces before deposition of electrodes. At least 2800 angstroms of surface material should be removed.

2. To prevent interelectrode diffusion bonding between platinum electrodes on adjacent wafers in a stacked and compressed pyrolytic boron nitride capacitor assembly, approximately 500 angstroms of radio frequency sputtered boron nitride should be deposited on the electrodes. Sputtered platinum electrodes approximately 3500 angstroms thick should be used rather than gold electrodes to obtain the best overall electrical performance at elevated temperature in vacuum.

3. A hermetically packaged, stacked pyrolytic boron nitride capacitor assembly consisting of ten or more wafers under compressive load (2 to 15 psi) should be tested under thermal aging and cycling conditions. Performance should be compared with that obtained for one and three wafer capacitors.

4. The feasibility of fabricating larger area (2 to 5 inches square) pyrolytic boron nitride capacitors should be determined. Mechanically lapped pyrolytic boron nitride wafers as well as "as deposited" boron nitride film should be evaluated.

5. Pyrolytic boron nitride capacitors should be tested under actual or simulated load conditions such as operation in a three-phase full wave rectifier assembly.

SECTION IV

PROGRAM III - EVALUATION, CONSTRUCTION AND ENDURANCE TESTING OF COMPRESSION SEALED PYROLYTIC BORON NITRIDE SLOT INSULATION

SUMMARY OF TECHNICAL RESULTS

The purpose of Program III was to determine if heat conductance from electrical conductors to the magnetic lamination stack could be improved at the conductor-insulation and insulation-slot interfaces. The means of accomplishing this has been reported in detail in reference 5 in which pyrolytic boron nitride was used as rigid slot insulation in a test statorette. Testing was performed at 1350° to 1450° F in a vacuum environment. In addition, pyrolytic boron nitride specimens were prepared and exposed to potassium vapor at 1400° F for 250 hours to determine the metal's effect on electrical breakdown strength. Specimens with test orientations in both the "a" and "c" axes were included (ref. 5).

Pyrolytic boron nitride slot insulation strips were oriented in the slots of specially designed statorettes to take advantage of the high thermal expansion rate in one axis and the high thermal conductivity in a second axis. Calculations were made to determine three different slot clearance and/or compression conditions in the 1350° to 1450° F test range. The associated room temperature slot clearance for each calculation was also determined. Three statorette assemblies were constructed and tested in sequence, based on the room temperature slot clearance required to meet each calculated condition.

Each assembly was then individually tested in a thermal vacuum chamber up to 1450° F at a vacuum in the 10^{-8} range. A temperature gradient was maintained from the statorette conductors to the laminations by supplying up to 140 amperes per winding to develop Joule (I^2R) heating. Data from testing the three assemblies showed that increasing the slot insulation contact area at the slot and conductor interfaces, by reducing clearance at the operating temperature did improve heat transfer by increased conductance. A statorette with conductors and insulation in place is shown in figure 7.

Electrical properties at room temperature for both "a" and "c" direction pyrolytic boron nitride wafers is reported in table 7. A high degree of stability is demonstrated by the data.

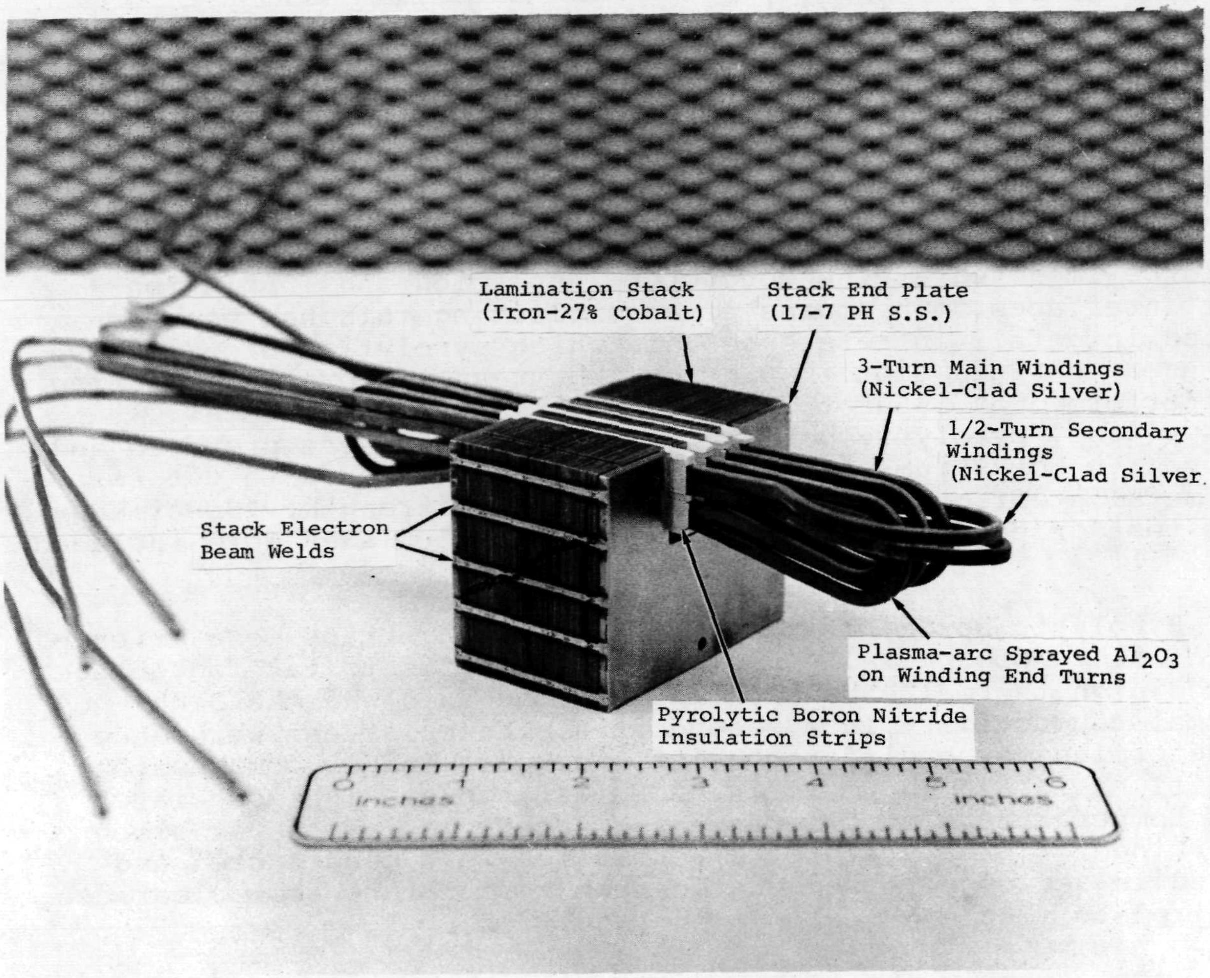

Figure 7. - Photograph of Pyrolytic Boron Nitride Insulated Statorette Prior to First Evaluation Test

Table 7. - Electrical Properties and DC Breakdown Voltage of Pyrolytic Boron Nitride Wafers Before and After Exposure to Potassium Vapor for 250 Hours at 1400° F.

Sample Group (3 specimens)		Orientation of Sputtered Platinum Electrodes(1)	Wafer Thickness (inches)	Potassium Exposure	Room Temperature Electrical Properties in Vacuum (1×10^{-6} torr)			
					DC Resistance @ 500 Vdc (ohms)	DC Breakdown Voltage	DC Breakdown Voltage (volts/mil)	Average DC Breakdown Voltage for 3 Specimens (volts/mil)
A	1	"c"	0.0016	None	$> 5 \times 10^{15}$	13,000	8100	9333
	2	"c"	0.0013	None	$> 5 \times 10^{15}$	12,000	9200	
	3	"c"	0.0013	None	$> 5 \times 10^{15}$	14,000	10,700	
B	1	"c"	0.0020	250 hours @ 1400°F	5×10^{11}	5100	2550	4176
	2	"c"	0.0013	250 hours @ 1400°F	1.3×10^{12}	6300	4750	
	3	"c"	0.0013	250 hours @ 1400°F	1.2×10^{12}	6800	5230	
C	1	"a"	0.0111	None	1×10^{15}	9700	873	> 916
	2	"a"	0.0103	None	2.5×10^{15}	> 12,000	> 1170	
	3	"a"	0.0102	None	2×10^{15}	7200	706	
D	1	"a"	0.0096	250 hours @ 1400°F	4.17×10^{11}	8200	853	1131
	2	"a"	0.0108	250 hours @ 1400°F	5×10^{11}	15,400	1430	
	3	"a"	0.0108	250 hours @ 1400°F	4.5×10^{11}	12,000	1110	

(1) Orientation: "c" direction; electrodes are parallel to plane of deposition of pyrolytic boron nitride
"a" direction; electrodes are perpendicular to plane of deposition of pyrolytic boron nitride

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

1. Three statorette constructions were successfully assembled and tested. However, the complexities encountered in assembling overlapping windings while maintaining extremely low slot interface clearances indicated that in a full size stator, winding end turn joining after assembly should be considered.

2. Thermal conductance from the conductors to the lamination stack was improved at operating temperature by a reduction of clearances in the conductor-insulation and insulation-lamination interfaces.

3. A zero clearance or compression fit across the various slot interfaces was required at operating temperature to obtain a low temperature difference from conductors to laminations.

4. The first increasing-temperature cycle to operating temperature within the two statorettes which attained compression, caused an improvement in conductor straightness. This resulted in a larger temperature difference at lower temperatures during subsequent thermal cycles.

5. Pyrolytic boron nitride insulation performed satisfactorily to temperatures over 1400° F in a vacuum of 10^{-8} torr when used with nickel-clad silver cored conductors having no wrapped insulation.

6. Pyrolytic boron nitride insulation temperature capability extends to the temperature limits of presently available magnetic materials.

7. Pyrolytic boron nitride is relatively more easily machined and lapped in comparison with other ceramic electrical insulating materials.

8. Nickel plugs welded to the conductor nickel cladding were successfully used on winding lead ends to seal the conductor silver core from the test environment.

9. Pyrolytic boron nitride retained considerable electrical strength after 250 hours exposure to potassium at 1400° F.

Recommendations

1. It is recommended that for any future stator or statorette testing using pyrolytic boron nitride and minimum slot clearance, the first operation should be an increasing-temperature cycle to design temperature, to "set" the conductors and insulation in the slots.

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